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Lubrication

A Technical Publication Devoted to
the Selection and Use of Lubricants

This Issue



GREASE
ANALYSIS



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GREASE ANALYSIS

THE petroleum testing manual of the American Society for Testing Materials defines "Lubricating grease — a solid to semi-fluid product of dispersion of a thickening agent in a liquid lubricant. Other ingredients imparting special properties may be included." Though fluid lubricants are used in greater volumes, it should be remembered that in nearly every machine there are critical areas where a semi-solid lubricant provides the optimum in lubrication.

In this connection the basic reason for use of the plastic-like greases is that they "stay put" in the space for which they are intended. Lubricating greases usually can be applied by simpler, less costly apparatus than that required for liquid lubricants.

The scope of this discussion is limited to the ordinary laboratory tests which generally are performed on greases. Physical and chemical procedures are described in the first part, and the significance of these tests to various phases of grease technology is considered in the latter discussion.

A summary of the grease procedures and their significance is given in Table I. Table III center-spread summarizes the general characteristics of eight groups of greases and shows typical results obtained from several of the tests described in this paper.

TESTS

Appearance and Odor

Although there is no standard procedure for

odor and color, these and other visual impressions are very helpful in identifying a grease. These observations may also show contamination, gross instability or non-conformity in manufacture. The color of a grease is sometimes enhanced by the addition of a dye.

Texture

One or more of the following terms generally describe grease texture: buttery, smooth, fibrous, stringy, firm, rubbery or sticky. Service requirements generally dictate the desired texture, and it is achieved by choice of ingredients and by variation in manufacturing techniques. Figure 1 shows how greases differ in appearance. While certain textures are usually associated with particular soap bases, wide variations from normal texture can be achieved by changes in manufacture.

Dropping Point

The dropping point is defined as "the temperature at which the grease passes from a semi-solid to a liquid state." Dropping point has no direct bearing upon the service performance of a grease. In addition, some greases separate an oily phase on heating before the gross melting point is reached. In such cases the dropping point is really a bleeding point.

In the dropping point method illustrated in Figure 2 the thermometer is located just above a

TABLE I
LABORATORY METHODS FOR TESTING GREASES

Name of Test	ASTM Designation	Method Summary	Significance
Penetration	D-217-52T	Empirical estimation of the consistency of greases by measuring extent of penetration of a standard cone.	Penetration (worked) classifies unused greases into grades. Measures softening of used greases.
Dropping Point	D-566-42	Temperature ($^{\circ}\text{F}$.) at which grease drops from hole in standard size cup.	Indicates temperature above which a grease may not be expected to perform satisfactorily but has no direct bearing on the service performance of the grease.
Water	D-128-47	Xylol mixed with grease and distilled — water carried over with Xylol. Volume separated water measured.	Manufacturing control or measures extent of water contamination.
Ash	D-128-47	Sample is burned, ignited and residue weighed.	Method for estimating non volatile metallic compounds.
Fat	D-128-47	See Table IV.	Manufacturing control.
Free Alkali or Free Fatty Acid	D-128-47	Sample dispersed in 50/50 petroleum ether and alcohol — titrated with standard acid or base solution.	Manufacturing control. Free Fatty Acids may indicate degrees of oxidation of used greases.
Soap	D-128-47	See Table IV.	Manufacturing control — and is also of value in identifying unknown or used greases.
Filler	D-128-47	Grease insolubles determined by using suitable solvent.	Manufacturing control — may be adapted to measure insolubles (dirt, metal chips, etc.) in used greases.
Oil	D-128-47	See Table IV.	Manufacturing control. Measures change in composition of used greases. Separated oil may be identified by usual oil tests.
Glycerin	D-128-47	Glycerin in water extract after acid decomposition is oxidized by periodate to formic acid which is titrated with sodium hydroxide.	Manufacturing control. Indicates whether grease manufactured from fats or fatty acids.
Oxidation Stability	D-942-50	Sample oxidized in bomb in oxygen atmosphere — decrease in pressure indicates degree of oxidation.	Measure resistance of greases to oxidation when stored under static conditions for long periods. It is not intended to predict stability under dynamic service conditions nor stability in commercial containers.
Evaporation	D-972-51T	Sample placed in cell maintained at elevated temperature for 22 hours. Evaporation calculated as loss in sample weight.	Measures loss of oil from greases at 210° to 300°F . Generally used for greases where evaporation loss is a factor.

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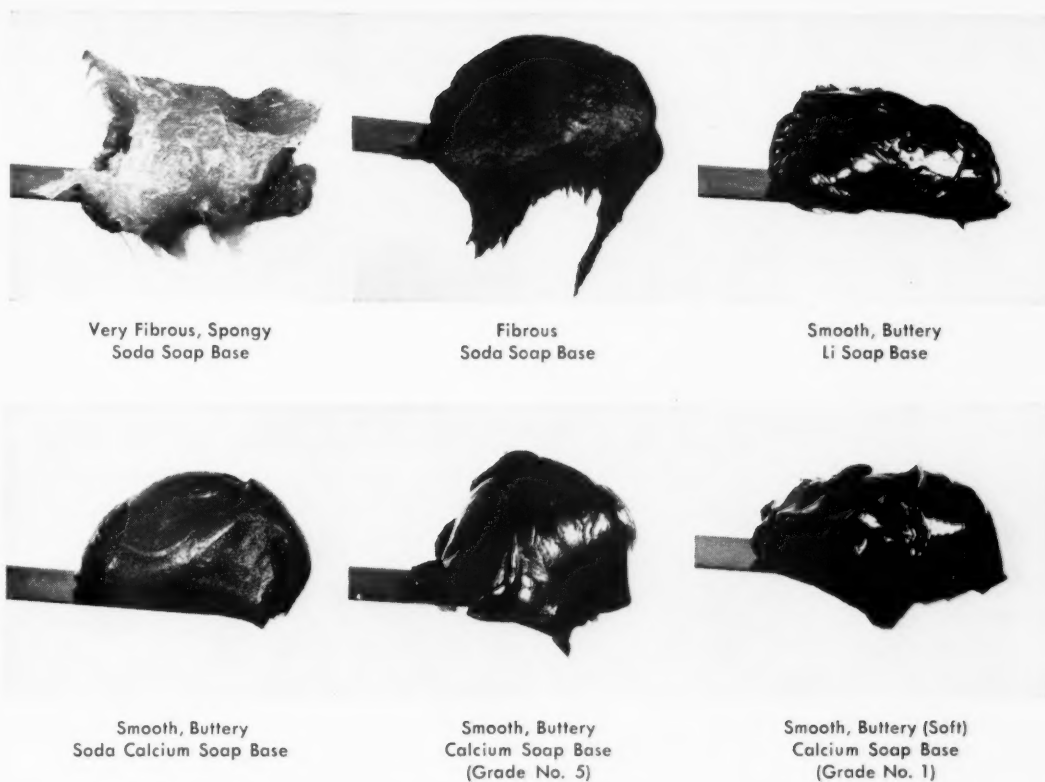


Figure 1 — Texture of Greases.

thin layer of grease which is placed in a chromium plated brass cup contained in a glass tube. The assembly is placed in an oil bath and heated at a specified rate until a drop of grease (or oily phase) falls through the orifice of the grease cup. The temperature at this point is noted and reported as the dropping point.

Some greases break down in operation near their dropping point while others may be used for a short period of time at temperatures above their dropping point. It is also significant as an indication of uniformity in manufacture and for purposes of identification. This test is not applicable to semi-fluid greases since they flow from the cup at room temperature.

Consistency — Penetration

Greases may vary in hardness from those which are nearly fluid at room temperature to those which are so hard that they can only be cut with a knife or wire. This property is defined as consistency and is undoubtedly the most frequently performed test on greases. They are classified in consistency ranges by penetration in much the same way that oils are classified by viscosity ranges.

Flow properties of greases differ considerably from those usually associated with oils. Most

greases are not deformed by small forces and under larger forces their rate of movement is not directly proportional to the force applied as is the case with petroleum oils.

Another very significant property of greases is that they may lose their consistency and become softer on working. This latter term refers to the movement of one layer of grease in relation to another layer which occurs whenever the grease is handled, stirred or otherwise undergoes movement as in a machine. Because of the change in consistency due to working, the measurements which most significantly correlate with service are those obtained after working.

The most commonly accepted method for measuring consistency is the cone penetration test described in ASTM Method D-217-52T. A sketch of the apparatus is shown in Figure 3. Penetration is measured by the depth, in tenths of a millimeter that a standard cone penetrates into the grease which is at 77°F.

The ASTM penetration of greases in original containers or removed from the container with a minimum handling is called "the Unworked Penetration". It is used for controlling manufacture and for determining the consistency of hard greases especially the brick type which cannot be worked easily.

TABLE II
N.L.G.I. CLASSIFICATION

N.L.G.I. Number	ASTM Worked Penetration
0	355-385
1	310-340
2	265-295
3	220-250
4	175-205
5	130-160
6	85-115

The ASTM test for Worked Penetration is the generally accepted method for measuring grease consistency. The ASTM Grease Worker is illustrated in Figure 4. To work a grease, the plunger is forced up and down through the grease for 60 double strokes. The worked penetration is then determined by use of the same standard cone as used for the unworked penetration. This procedure makes for maximum reproducibility, as all samples are worked to the same extent immediately before penetrating the sample.

The ASTM Worked Penetration test is often continued for as many as 100,000 strokes in order to indicate the shear resistance, or resistance to softening, of the grease. This has become important in recent years, as some critical applications, such as aircraft magneto gear boxes, cannot tolerate a grease which shears down badly on working. Modern research has developed products which show negligible softening on working, for applications where leakage is a serious problem.

The National Lubricating Grease Institute has developed a classification system for greases based on worked penetrations (60 strokes). As illus-

trated in Table II this allows only a 30 point range in penetration for each class with a 15 point gap between grades.

In following the performance of greases in service, it is often of interest to determine the penetration when only very small samples are available, as for example, on samples from ball and roller bearings. In such cases, a miniature penetrometer has been developed. This apparatus requires only three to five rather than four hundred to five hundred grams of grease and is illustrated in Figure 5.

Another method for determining penetrations on samples of grease which are too small for the standard ASTM penetrometer and worker is that employing the quarter scale cone. Results by this procedure correlate fairly well with those obtained by the full scale cone.

Consistency — Apparent Viscosity

Another physical test often called for in grease specifications is ASTM Method D-1092 "Apparent Viscosity of Lubricating Greases". A hydraulic system forces the grease through a capillary and the apparent viscosity is calculated from the flow rate and the force developed in the system. The apparatus is illustrated in Figure 6.

Apparent viscosity of a grease governs to a large extent the ease of handling and dispensing, as well as the starting and running torque of mechanisms that are grease lubricated. This test is carried out only on softer grades of grease.

Oil Bleeding

The separation of oils from lubricating greases is determined by a wide variety of procedures. In

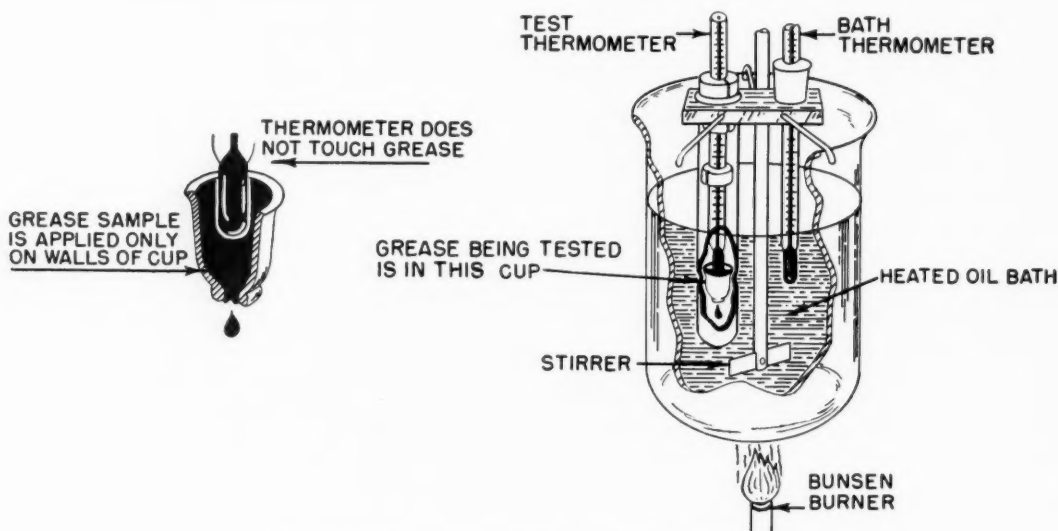


Figure 2 — Apparatus Used to Determine Dropping Point.



Figure 4 — ASTM Motorized Grease Worker.

one of the typical procedures, a 10 gram sample is weighed on a sixty mesh nickel gauze which has been shaped in the form of a cone. The cone is suspended in a beaker and placed in an oven for a prescribed temperature and interval of time. The oil which drips through the screen is weighed and its per cent calculated.

Considerable care should be used in interpreting oil separation tests since there is little data directly correlating these results with actual field performance. Obviously, gross instability is undesirable, but on the other hand, a small amount of oil bleeding is desirable since it insures immediate lubrication of parts when machinery is first started up.

Evaporation Loss

Another test of interest is the evaporation loss of lubricating greases. In this procedure, the grease is placed in an evaporation cell and heated air is passed over its surface for 22 hours. The per cent evaporation loss is calculated from the loss in weight of the sample. This method is primarily intended for low temperature greases made from oils of low viscosity. From a practical standpoint, it should be remembered that the test is carried out under static, rather than dynamic conditions with ever changing surface such as encountered in service.

Water Washout

Over the years there have been many tests developed to simulate the actual environment in

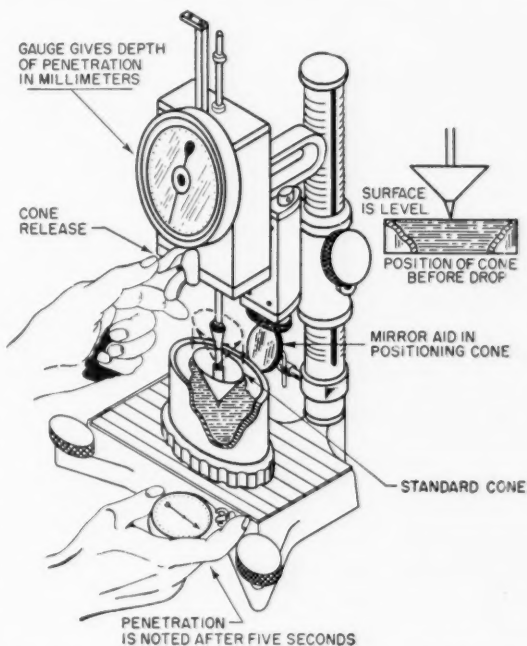


Figure 3 — This Drawing Shows ASTM Penetration Apparatus.

TABLE III
GENERAL CHARACTERISTICS

<u>Soap Base</u>	<u>Texture</u>	<u>Dropping Point (Approx.)</u>	<u>Condition After Heating Above Melting Point & Cooling</u>	<u>Maximum Continuous Useable Temperature</u>	<u>Efficiency of V</u>
<u>Calcium-General</u>	Buttery	200-225°F.	Separates; when properly chemically stabilized has non-separating properties.	175°F.	Res
<u>Calcium Resinate</u>	Buttery	275°F.	Foams at 200°F. Separates on prolonged heating.	200°F.	Res
<u>Sodium General</u>	Fibrous or Smooth	300-450°F.	No change if worked	300-400°F.	Sus
<u>Sodium Brick Type</u>	Hard-Brittle	300-450°F.	No change	300-450°F.	Sus
<u>Aluminum</u>	Some Buttery. Some stringy but never fibrous	200°F.	Changes texture upon cooling but does not separate above melting point or below.	200°F. (See remarks)	Res
<u>Lithium</u>	Buttery to Stringy	360°F.	No change if worked	300°F.	Res
<u>Mixed Base Sodium-Calcium</u>	Fairly Buttery to fibrous	315°F.	No change if worked	250°F.	Sus
<u>Solid Thickened Greases</u>	Smooth Buttery to slightly stringy	500 Plus	May harden after heating, but retains grease texture.	Depends on oil component.	Res
<u>High-Melting Point Calcium</u>	Buttery	280°F.	Separates	225°F.	Res

TABLE III

CHARACTERISTICS OF GREASES

<u>Effect of Water</u>	<u>Resistance to Softening Upon Working</u>	<u>Primary Use</u>	<u>Remarks</u>
Resistant	Fair to Poor	General purpose industrial lubricant for plain bearing and line shafting.	Calcium soap greases stabilized with high boiling chemicals instead of water have higher dropping points and can be used to higher maximum temperatures. They return to a homogeneous state upon cooling after being melted.
Resistant	Fair to Poor	Rough heavy bearings operating at slow speeds, also skids, track curves, and wagon wheels.	Usually used where low cost is a major factor.
Susceptible	Excellent to Poor (Varies greatly with composition)	Ball and roller bearings operating at low to medium speeds, and light to heavy loads; also on wheel bearings and chassis of automobiles and trucks.	Susceptibility to water can be improved by certain choices in composition. Provides rust protection. Short fibered or smooth textured greases used on anti-friction bearings, while long fibered greases recommended for chassis lubrication.
Susceptible	Fair to Poor (Varies greatly with composition)	Driving journals of locomotives and similar bearings lubricated with brick greases.	Usually applied directly against revolving journal.
Resistant	Fair to Poor	Special applications where resistance to centrifugal force or adhesiveness is desired.	These greases change texture upon heating and upon cooling. Therefore, they are not recommended for use above 150°F.
Resistant	Excellent to Poor (Varies greatly with composition)	Aircraft lubrication at temperatures from -100°F. to +300°F. and many applications in automotive and industrial use.	The combination of water resistance with high dropping point is advantageous for many applications over a wide range of temperatures.
Susceptible	Excellent to Poor (Varies greatly with composition)	All types ball and roller bearings, and special applications at both high and low temperatures depending upon composition.	Mixed base greases are a combination of two or more types of greases. The most outstanding example is sodium-calcium soap base greases, properties of which resemble characteristics of major component.
Resistant	Excellent to Poor	High temperature applications.	Solid thickeners may be finely divided Clay, Clay derivatives, Silica or Carbon Black.
Resistant	Excellent	Ball, roller and plain bearings in automotive use including wheel bearings and chassis; and in industrial plants.	High melting point stabilized calcium base greases have inherent rust proofing properties. They fill the gap between calcium general and sodium base greases.

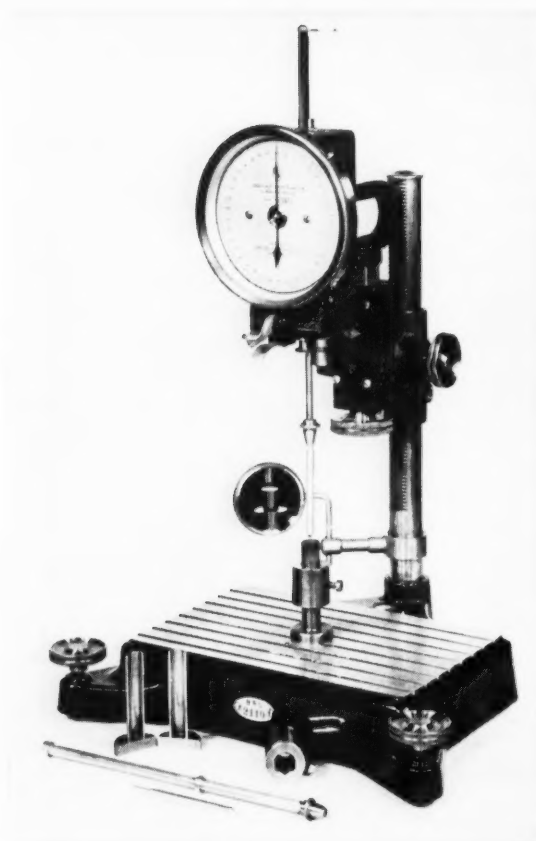


Figure 5 — Miniature Penetrometer for Very Small Samples.

which a grease might find itself in service. One of the few such tests which have received official recognition is ASTM D-1264 entitled, "Water Washout Characteristics of Lubricating Greases". Four grams of grease are contained in a ball bearing which is placed in a prescribed housing and rotated at 600 ± 30 r.p.m. Water at either 100 or 175°F. impinges on the bearing housing at a rate of 5 ± 0.5 ml. per second. The amount of grease washed away in one hour is to some degree a measure of its resistance to water washout, although the results of this test may not agree with those in service because of housing or seal design.

Particle Content

Federal Specification VV-L-791e Method 3005.1 determines the size in microns and concentrations of foreign particles in lubricating greases. This is done by actually measuring the particles in the grease placed in a counting chamber of known volume. A compound microscope capable of approximately 60 diameters magnification is employed and the number of particles per cubic centimeter are reported according to size classes. Though the interpretation as to what constitutes

a foreign particle is in part left up to the operator, the precision of this method is fairly good. Most specifications allow a few thousand particles in the range of 25 microns or larger. However, one particle larger than 125 microns would generally disqualify a grease.

ASTM has published for information a "Proposed Method of Test for Estimation of Deleterious Particles in Lubricating Grease" which is based on the number of scratches caused by such particles on a polished plastic surface. In this fairly rapid procedure, the sample is placed between two clean highly polished acrylate plastic plates which are then rotated about 30° with respect to each other. The number of characteristic arc-shaped scratches give a measure of the deleterious particles. The significance of the number of scratches and particle counts has not been directly correlated with field performance.

Corrosion

Federal Specification VV-L-791e Method 5309.1 measures the corrosiveness of greases to copper by use of a highly polished copper strip about 1/2 inch in width and 3 inches in length which is partly immersed in a sample of grease. The grease is placed in an oven at 212°F. for a specified number of hours, allowed to cool to room temperature, and the copper strip washed with benzene and examined. The portion of the copper strip which had been in contact with the grease is compared with that exposed to the air during the test. The degree of pitting, etching or staining of the strip is reported as well as any change in appearance of the grease.

ASTM has a method for establishing the effect of grease on copper (D-1261). It involves immersion of a prepared copper strip in the grease which is then subject to oxygen pressure in a bomb at

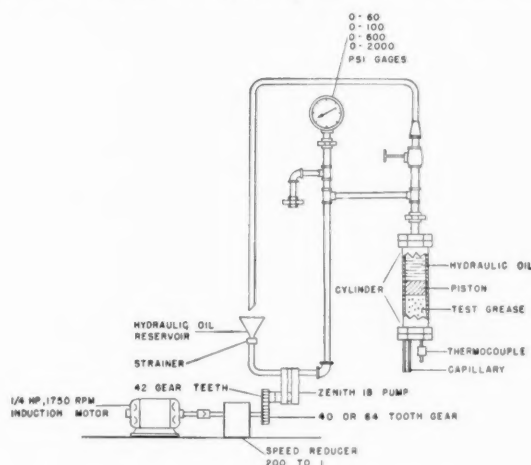


Figure 6 — Drawing of ASTM Apparatus for Apparent Viscosity.



Figure 7 — Oxidation Stability Apparatus.

210°F. The appearance of the copper strip is used as the measure of corrosion.

Oxidation

A procedure frequently employed for determining the oxidation stability of greases is ASTM Method D 942. The apparatus consists of an oxidation bomb assembly illustrated in Figure 7 and an oil bath capable of maintaining a temperature between 209 and 211°F. Four grams of grease are placed in each of five glass dishes held in a rack and this assembly is placed in the bomb which is then filled with oxygen at 110 p.s.i. The oxygen pressure drop in a specified time is a measure of the lubricant's resistance to oxidation under static conditions.

Generally a grease exhibits two stages of oxidation, the first one is relatively slow and termed the "induction period". The second stage is evidenced by a sharp decrease in oxygen pressure during which time the properties of the grease change.

The greatest significance of this test is that it predicts the tendency of a grease to oxidize on ball and roller bearings during shelf storage, but no correlation exists with grease stability under dynamic conditions such as exist in actual bearing

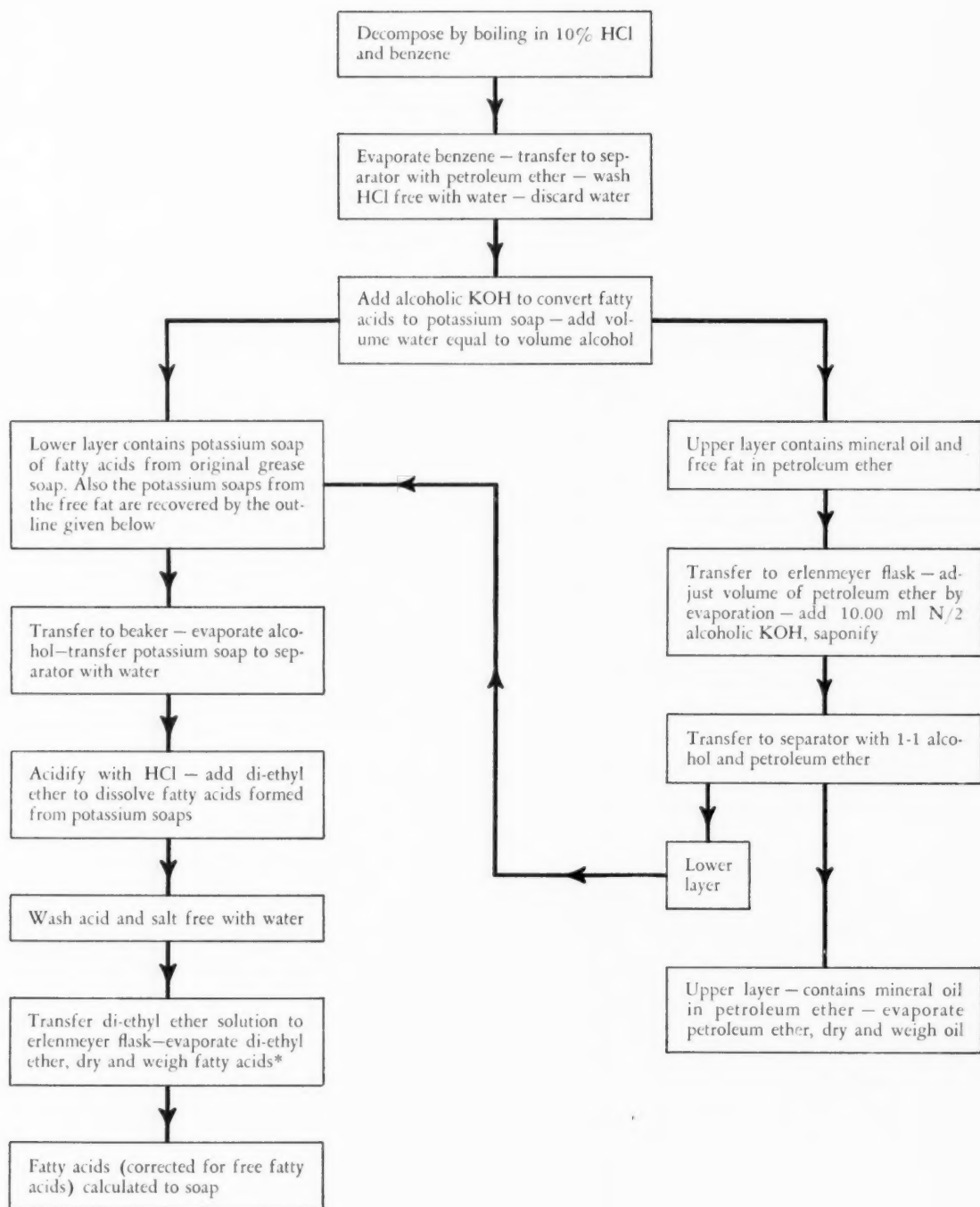
service. This procedure will broadly differentiate between greases in respect to oxidation stability and will generally indicate whether the grease contains an oxidation inhibitor. A good uninhibited grease may show a pressure drop of 50 + p.s.i. as compared to a drop of 5 p.s.i. in 100 hours for a good inhibited grease.

Chemical Analysis

ASTM Method D-128 gives procedures for determining the following components of a grease: soap, unsaponifiable matter (petroleum oil), water, free alkali, free fatty acid, fat, glycerine and filler. The method of breaking down a grease for analysis is graphically shown in Table IV. Method D-128 does not apply to greases made with non-soap thickeners nor to those which contain other than petroleum oils. Though the major constituents of the grease are petroleum oil and metallic soap, there may be present in the grease many other components.

Strictly from a performance angle, analysis for the various constituents in the original grease is of little significance. However, the grease producer is very much interested in such information from

TABLE IV
SIMPLIFIED SCHEME OF GREASE ANALYSIS
METHOD ASTM D 128-47



*Stop at this point for free fat analysis.

the standpoint of manufacturing control and for technical service work. Analyses of used greases are very helpful in measuring the extent of deterioration in service, and in detecting contamination as discussed in the following sections.

SIGNIFICANCE OF GREASE TESTING

The testing of greases can be logically and conveniently considered in relation to three phases of petroleum technology. These are:

1. Research and Development
2. Manufacture and Control
3. Product Application

Although laboratory tests are used to predict characteristics of greases, the final criterion of a satisfactory grease is its actual performance in use.

Research and Development

Research and Development in connection with greases is concerned with developing new greases, improving existing ones and developing fundamental information on their behavior. In research work, especially if new aspects are being investigated, accepted methods may not be applicable; therefore new tests and techniques must be designed.

Although some aspects of research are referred to as "pure research", the ultimate aim of any program is to obtain information that will eventually be useful in developing products giving better performance.

For example, it was known in the late 1930's, through the use of the optical microscope, that many grease soaps were fibrous. Rough estimates had been made of the lengths of some of these fibers and correlations were drawn between the estimated fiber lengths and the consistency of the parent grease. In these studies, however, nothing was learned of the width, thickness or surface structure of the fibers.

During the last decade tremendous progress has been made, through the use of the electron microscope, in the characterization of soap structures and in the correlation of these structures to the physical properties and behavior of the greases. Early estimates of length were confirmed. Widths and thickness have been accurately measured. Differences in surface textures have been discovered.

We now use such terms as hank-of-hair, two-strand rope, and twisted ribbon to describe the various ways in which the soap micelles or fibrils get together to make the familiar soap fibers.

The immediate and practical purpose of research and development is to formulate a grease that provides satisfactory lubrication for a certain type of application. For example, research might be

directed to the development of a grease for high temperature applications. Besides the requirement of satisfactory performance at high temperatures, the development might also be guided by other service requirements and by a customer specification. Merely meeting a specification which covers laboratory tests does not guarantee satisfactory field performance. Only service performance can be conclusive.

During the development of a new grease, or improving an existing formula, much experimental work is necessary to provide an acceptable formulation of a grease. The grease undergoes extensive and complicated laboratory bench testing simulating the most severe conditions to be encountered in actual service.

A formulation that is thought to have the desired performance characteristics, as indicated by laboratory and bench tests, is then ready for actual field trials. If the formulation meets expectations and gives satisfactory field performance, it takes its place with other regularly marketed greases.

Although work may be directed towards developing a grease for a specific application, the experimental formulations may be found to have other exceptional properties.

Manufacture and Control

After the satisfactory nature of a formulation has been established, a relatively few laboratory tests will generally suffice to insure that succeeding batches will have the same characteristics as the prototype. The following tests are usually sufficient to insure continued uniformity in manufacture:

1. Penetration
2. Dropping Point
3. Water
4. Free Fatty Acid or Free Alkali
5. Soap Content
6. Oil Content

Batches of greases intended for special or severe service applications are evaluated by additional control tests which are especially applicable. For example, a grease manufactured to have exceptional oxidation stability would most likely be required to meet a minimum pressure drop in the oxygen bomb test.

Product Application

Testing of greases does not stop after a new grease has been placed on the market. The manufacturer not only follows a new product until he is satisfied that it is completely satisfactory but also periodically checks established products to be

sure that quality is maintained. In addition, problems that arise in the field can oftentimes be solved by laboratory tests and examinations. However, laboratory examinations will not always disclose improper maintenance conditions, such as improper bearing packing. Greases, as well as oils, become oxidized, contaminated, and change in physical characteristics in service.

Testing samples of grease from the field has several objectives.

First: To establish the identity of the product. Since many greases may have the same general appearance, it is usually necessary to analyze the sample for its components such as soap, mineral oil, and filler. While appearance and physical tests of a grease are usually helpful in establishing identity, greases may be so radically changed through contamination and use that only tests on component parts are of significance. It is often possible to determine if two greases have been mixed in service provided tests on the component parts of each grease are known. The identification of used greases is frequently required to establish whether the proper grease for the particular service has been employed.

Second: To establish the degree and nature of contamination. Contamination occurs in two ways—(1) accidental mixing into the grease of foreign materials that would be detrimental to the grease or machinery and (2) contamination by wear products, dirt, dust or other environmental material. Since the first type of contamination could include almost anything, only the skill and experience of the analyst can be relied on to discover its nature. Abrasives, drying oils, and paints come to mind as examples of such contamination. Fortunately such contamination is rare. Since most industrial applications are in environments containing dust and dirt, these contaminants are quite common in used greases. Wear metals also are retained in the greases. Since greases cannot be filtered or purified, contaminants must be removed by relubrication to purge the system of the contaminated grease along with the contaminants.

Usually the contaminants are of a solid nature. Recovering them from a used grease for inspection is accomplished by use of suitable solvents. For example, dirt, dust, wear metals and miscellaneous trash could be separated from a grease by extracting with a mixture of benzene and ethyl alcohol. One or a combination of microscopic, spectrographic, and X-ray examinations should disclose the nature of the insolubles.

The form in which the contaminants are found is of prime importance. The presence of alpha ferric oxide (Fe_2O_3) would, in itself, be strong evidence that fretting corrosion had occurred. The

identification of a black iron compound as Fe_3O_4 would indicate a rusting condition of the bearing metals or of metallic particles originating from wear. The presence of silica is an indication that the grease is contaminated with dirt or dust.

The amount of contamination is also important. In any piece of machinery wear is bound to occur. The presence of small amounts of wear metals in used greases is not considered unusual. Extraordinary amounts usually indicate malfunctioning equipment or improper maintenance. An obvious effect of solid contaminants is the discoloration of the grease. Actually it takes only a very small amount of dirt and wear particles to discolor a light-colored product whereas dark colored greases are frequently little changed by gross contamination.

Third: To determine whether the essential characteristics necessary for continued use are still retained by the grease. The analyst must make certain that the structure of the grease has been maintained and that the grease has not become excessively oxidized or contaminated. Since used greases cannot be purified in service, they must be removed to eliminate contaminants and degradation products. Usually, by suitable tests, safe relubrication periods can be established which, if followed, obviate repeated testing of the used grease and insure against machine failures.

Fourth: To resolve field problems and establish reasons for unsatisfactory performance. This involves selecting suitable tests and correct interpretation of test data. In order to do this one must have a precise knowledge of the operating conditions, maintenance practices and of the characteristics of greases involved. A simple example is the case where it is reported that a grease separates in use in an application operating at about 225°F. Investigation might disclose that the grease being used is a water stabilized cup grease which separates into soap and oil at temperatures above 175°F. In this case it is obvious that the wrong type of grease was used.

CONCLUSIONS

In selecting laboratory tests to be run on either used or unused greases, only those tests which will give information having a bearing on the problem are worthwhile. Consideration must be given to the type of lubricant, its general nature and composition as well as the application involved. Before marketing a new grease product, a reliable and competent manufacturer evaluates it, not only by chemical, physical, and bench tests but also by extensive field performance tests which include the most severe conditions of operation to which the grease is likely to be subjected.

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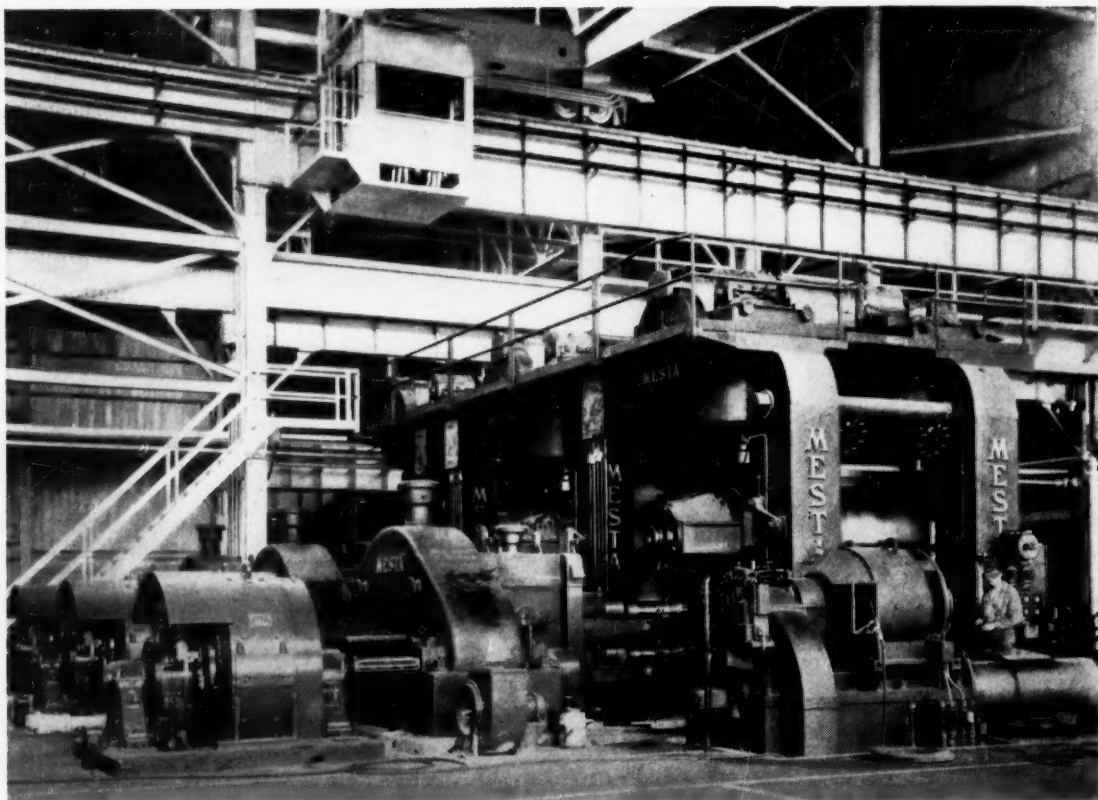
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